

We claim:

- 1 1. A polymorphic form of 9-nitrocamptothecin, the polymorph being
2 characterizable as having, by differential scanning calorimetry, no observable
3 endotherm and an exotherm at between 273.6 and 275.6 °C, and a solution NMR
4 spectrum with multiplets at 1.7 and 3.7 ppm shifts.
- 1 2. A polymorphic form of 9-nitrocamptothecin according to claim 1, the
2 polymorph being further characterizable as having an exotherm by differential
3 scanning calorimetry at between 274.1 and 275.1 °C.
- 1 3. A polymorphic form of 9-nitrocamptothecin according to claim 1, the
2 polymorph being further characterizable as having an exotherm by differential
3 scanning calorimetry at between 274.4 and 274.8 °C.
- 1 4. A polymorphic form of 9-nitrocamptothecin according to claim 1, the
2 polymorph being further characterizable as having an exotherm by differential
3 scanning calorimetry at between 274.5 and 274.7 °C.
- 1 5. A polymorphic form of 9-nitrocamptothecin according to claim 1, wherein the
2 polymorph is obtained by grinding.
- 1 6. A polymorphic form of 9-nitrocamptothecin, the polymorph being
2 characterizable as having an X-ray powder diffraction pattern with diffraction lines at
3 2θ values 6.7, 12.5, 14.0 and 23.9 for Cu $K\alpha$ radiation of wavelength 1.5406
4 Angstrom.

1 7. A polymorphic form of 9-nitrocamptothecin, the polymorph being
2 characterizable as having an X-ray powder diffraction pattern with diffraction lines at
3 $^{\circ}2\theta$ values 6.7, 12.5, 14.0 and 23.9 for Cu $K\alpha$ radiation of wavelength 1.5406
4 Angstrom.

1 8. A polymorphic form of 9-nitrocamptothecin, the polymorph being
2 characterizable as having, for Cu $K\alpha$ radiation of wavelength 1.5406 Angstrom, an X-
3 ray powder diffraction pattern with diffraction lines at $^{\circ}2\theta$ values 6.7, 12.5, 14.0 and
4 23.9.

1 9. 9-nitrocamptothecin in a form crystallized from tetrahydrofuran.

1 10. A polymorphic form of 9-nitrocamptothecin according to claim 10, the
2 polymorph being characterizable as having, by differential scanning calorimetry, no
3 observable endotherm and an exotherm at between 273.6 and 275.6 $^{\circ}\text{C}$, and a solution
4 NMR spectrum with multiplets at 1.7 and 3.7 ppm shifts.

1 11. A polymorphic form of 9-nitrocamptothecin according to claim 10, the
2 polymorph being characterizable as having an X-ray powder diffraction pattern with
3 diffraction lines at $^{\circ}2\theta$ values 6.7, 12.5, 14.0 and 23.9 for Cu $K\alpha$ radiation of
4 wavelength 1.5406 Angstrom.

1 12. A polymorphic form of 9-nitrocamptothecin according to claim 10, the
2 polymorph being characterizable as having an X-ray powder diffraction pattern with
3 diffraction lines at $^{\circ}2\theta$ values 6.7, 12.5, 14.0 and 23.9 for Cu $K\alpha$ radiation of
4 wavelength 1.5406 Angstrom.

1 13. A polymorphic form of 9-nitrocamptothecin according to claim 10, the
2 polymorph being characterizable as having, for Cu $K\alpha$ radiation of wavelength 1.5406

3 Angstrom, an X-ray powder diffraction pattern with diffraction lines at $^{\circ}2\theta$ values 6.7,
4 12.5, 14.0 and 23.9.

1 14. A pharmaceutical composition comprising:
2 a pharmaceutical carrier; and
3 a polymorphic form of 9-nitrocamptothecin, the polymorph being
4 characterizable as having, by differential scanning calorimetry, no observable
5 endotherm and an exotherm at between 273.6 and 275.6 $^{\circ}\text{C}$, and a solution NMR
6 spectrum with multiplets at 1.7 and 3.7 ppm shifts.

1 15. A pharmaceutical composition according to claim 14, the polymorph being
2 further characterizable as having an exotherm by differential scanning calorimetry at
3 between 274.1 and 275.1 $^{\circ}\text{C}$.

1 16. A pharmaceutical composition according to claim 14, the polymorph being
2 further characterizable as having an exotherm by differential scanning calorimetry at
3 between 274.4 and 274.8 $^{\circ}\text{C}$.

1 17. A pharmaceutical composition according to claim 14, the polymorph being
2 further characterizable as having an exotherm by differential scanning calorimetry at
3 between 274.5 and 274.7 $^{\circ}\text{C}$.

1 18. A pharmaceutical composition comprising:
2 a pharmaceutical carrier; and
3 a polymorphic form of 9-nitrocamptothecin, the polymorph being
4 characterizable as having an X-ray powder diffraction pattern with diffraction lines at
5 $^{\circ}2\theta$ values 6.7, 12.5, 14.0 and 23.9 for Cu $K\alpha$ radiation of wavelength 1.5406
6 Angstrom.

1 19. A pharmaceutical composition comprising:
2 a pharmaceutical carrier; and
3 a polymorphic form of 9-nitrocamptothecin, the polymorph being
4 characterizable as having an X-ray powder diffraction pattern with diffraction lines at
5 $^{\circ}2\theta$ values 6.7, 12.5, 14.0 and 23.9 for Cu $K\alpha$ radiation of wavelength 1.5406
6 Angstrom.

1 20. A pharmaceutical composition comprising:
2 a pharmaceutical carrier; and
3 a polymorphic form of 9-nitrocamptothecin, the polymorph being
4 characterizable as having, for Cu $K\alpha$ radiation of wavelength 1.5406 Angstrom, an X-
5 ray powder diffraction pattern with diffraction lines at $^{\circ}2\theta$ values 6.7, 12.5, 14.0 and
6 23.9.

1 21. A pharmaceutical composition comprising:
2 a pharmaceutical carrier; and
3 a polymorphic 9-nitrocamptothecin in a form crystallized from
4 tetrahydrofuran.

1 22. A pharmaceutical composition according to claim 21, the polymorph being
2 characterizable as having, by differential scanning calorimetry, no observable
3 endotherm and an exotherm at between 273.6 and 275.6 $^{\circ}\text{C}$, and a solution NMR
4 spectrum with multiplets at 1.7 and 3.7 ppm shifts.

1 23. A pharmaceutical composition according to claim 21, the polymorph being
2 characterizable as having an X-ray powder diffraction pattern with diffraction lines at
3 $^{\circ}2\theta$ values 6.7, 12.5, 14.0 and 23.9 for Cu $K\alpha$ radiation of wavelength 1.5406
4 Angstrom.

1 24. A pharmaceutical composition according to claim 21, the polymorph being
2 characterizable as having an X-ray powder diffraction pattern with diffraction lines at
3 $^{\circ}2\theta$ values 6.7, 12.5, 14.0 and 23.9 for Cu $K\alpha$ radiation of wavelength 1.5406
4 Angstrom.

1 25. A pharmaceutical composition according to claim 21, the polymorph being
2 characterizable as having, for Cu $K\alpha$ radiation of wavelength 1.5406 Angstrom, an X-
3 ray powder diffraction pattern with diffraction lines at $^{\circ}2\theta$ values 6.7, 12.5, 14.0 and
4 23.9.

1 26. A method of preparing a polymorphic form of 9-nitrocamptothecin, the
2 method comprising:
3 crystallizing 9-nitrocamptothecin from tetrahydrofuran.

1 27. A method according to claim 26, the polymorph being characterizable as
2 having, by differential scanning calorimetry, no observable endotherm and an
3 exotherm at between 273.6 and 275.6 $^{\circ}\text{C}$, and a solution NMR spectrum with
4 multiplets at 1.7 and 3.7 ppm shifts.

1 28. A method according to claim 26, the polymorph being characterizable as
2 having an X-ray powder diffraction pattern with diffraction lines at $^{\circ}2\theta$ values 6.7,
3 12.5, 14.0 and 23.9 for Cu $K\alpha$ radiation of wavelength 1.5406 Angstrom.

1 29. A method according to claim 26, the polymorph being characterizable as
2 having an X-ray powder diffraction pattern with diffraction lines at $^{\circ}2\theta$ values 6.7,
3 12.5, 14.0 and 23.9 for Cu $K\alpha$ radiation of wavelength 1.5406 Angstrom.

1 30. A method according to claim 26, the polymorph being characterizable as
2 having, for Cu $K\alpha$ radiation of wavelength 1.5406 Angstrom, an X-ray powder
3 diffraction pattern with diffraction lines at $^{\circ}2\theta$ values 6.7, 12.5, 14.0 and 23.9.